

Cyanide Destruction/ Immobilization of Residual Sludge

Mixed Waste Focus Area



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Cyanide Destruction/ Immobilization of Residual Sludge

OST Reference # 1425

Mixed Waste Focus Area



Demonstrated at
U.S. Department of Energy
Rocky Flats Environmental Technology Site
Golden, Colorado



Purpose of this document

Innovative Technology Summary Reports are designed to provide potential users with the information they need to quickly determine if a technology would apply to a particular environmental management problem. They are also designed for readers who may recommend that a technology be considered by prospective users.

Each report describes a technology, system, or process that has been developed and tested with funding from DOE's Office of Science and Technology (OST). A report presents the full range of problems that a technology, system, or process will address and its advantages to the DOE cleanup in terms of system performance, cost, and cleanup effectiveness. Most reports include comparisons to baseline technologies as well as other competing technologies. Information about commercial availability and technology readiness for implementation is also included. Innovative Technology Summary Reports are intended to provide summary information. References for more detailed information are provided in an appendix.

Efforts have been made to provide key data describing the performance, cost, and regulatory acceptance of the technology. If this information was not available at the time of publication, the omission is noted.

All published Innovative Technology Summary Reports are available online at <http://em-50.em.doe.gov>.

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SECTION 1

Technology Summary

Metal-plating operations at U.S. Department of Energy (DOE) facilities have generated wastes contaminated with cyanide. Alkaline chlorination is the most widely used method for destruction of cyanide in plating and other chemical wastes. However, the process uses an external source of chlorine under high-pH conditions to chemically destroy cyanide complexes, producing nontoxic chlorides, carbonates, and hydroxides. A disadvantage of this method is that it requires the purchase and storage of large quantities of hazardous chlorine gas or hypochlorite solutions. Electrochemical chlorination is an innovative method of cyanide destruction that reduces the safety risks associated with these chlorine sources.

Electrochemical chlorination uses an electrolytic cell to produce chlorine gas from sodium chloride (NaCl) in situ, thereby eliminating safety hazards associated with on-site storage of chlorine gas. Sodium chloride is added to the cyanide waste solution as the chlorine source. The waste is circulated through an electrolytic cell equipped with inert platinum anodes. The electrodes generate powerful oxidizing agents (e.g., chlorine gas and hydroxyl radicals) and convert the cyanide to carbon dioxide and nitrogen.

Demonstration Summary

Electrochemical chlorination was developed to safely treat cyanide waste streams classified as mixed low-level waste. The more conventional methods of cyanide destruction were unavailable for use at the Rocky Flats Environmental Technology Site (RFETS) due to safety concerns at the site. The technology was first demonstrated at RFETS in 1994–95 under a Resource Conservation and Recovery Act (RCRA) Treatability Study Exemption. It was further demonstrated in 1996 as part of the Mixed Waste Focus Area Quick Wins Program.

- RFETS first performed treatability studies using electrochemical chlorination on plating wastes and excess chemicals generated during plating operations, including a gas, solids, and solutions classified as low-level mixed wastes. The treatability study consisted of testing four different types of cyanide wastes: cadmium cyanide plating bath, gold cyanide plating bath, dilute cyanide plating rinse, and various excess chemical cyanides such as potassium cyanide, silver cyanide, and hydrogen cyanide. During the treatability study, over 800 L of cyanide waste was successfully treated and disposed of.
- Based on the success of the process in treatability studies, RFETS secured a RCRA Part B permit modification in March 1996 and completed treatment of an additional 1,130 L (six drums) of spent cyanide plating waste by November 1996. This activity eliminated the remaining known inventory of this waste type at RFETS. The technology and RCRA permit for the electrochemical chlorination unit are being maintained and operated by a subcontractor at RFETS for treatment of additional cyanide wastes as they are discovered.
- The electrochemical chlorination process is ready for implementation, based on its demonstrated success at RFETS. Experience and information gained from the RFETS work should be applicable to RCRA permit applications at other sites; site-specific variables will need to be considered, however.



Key Results

- Results demonstrate that the electrochemical chlorination technique is a simple, effective process capable of achieving the destruction of soluble cyanide chemicals in batches up to 190 L.
- Electrochemical chlorination is effective in achieving cyanide concentrations below the regulatory treatment standards.
- The electrochemical chlorination apparatus is easy to operate.
- No toxic hydrogen cyanide gas or cyanogen chloride was detected in the off-gas from the scrubber/vent system.
- Silver may interfere with current analytical methods and require modified methods.

Electrochemical chlorination has been successfully used to destroy cyanide in excess plating wastes and in excess chemicals at RFETS. The process has the potential to treat additional cyanide wastes at RFETS and cyanide waste streams at a number of other DOE sites, including Lawrence Berkley National Laboratory, Oak Ridge National Laboratory, Paducah Gaseous Diffusion Plant, Portsmouth Gaseous Diffusion Plant, and the Oak Ridge Y-12 Plant. Most of these waste streams represent small-quantity wastes with unique characteristics.

Contacts

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Licensing Information

All equipment and materials are commercially available.

Other

All published Innovative Technology Summary Reports are available online at <http://em-50.em.doe.gov>. The Technology Management System, also available through the EM50 Web site, provides information about OST programs, technologies, and problems. The OST Reference # for cyanide destruction is 1425.



SECTION 2

Overall Process Schematic

Alkaline chlorination is the most widely used cyanide destruction method. The process normally uses chlorine gas at high-pH conditions to chemically destroy cyanide complexes, producing reaction products such as chlorides, carbonates, and hydroxides. The electrochemical chlorination process was developed to avoid use of an external source of chlorine gas. The electrolytic cell used in the process generates the chlorine gas in situ. Figure 1 is a simplified engineering drawing of the electrochemical chlorination cyanide destruction process.

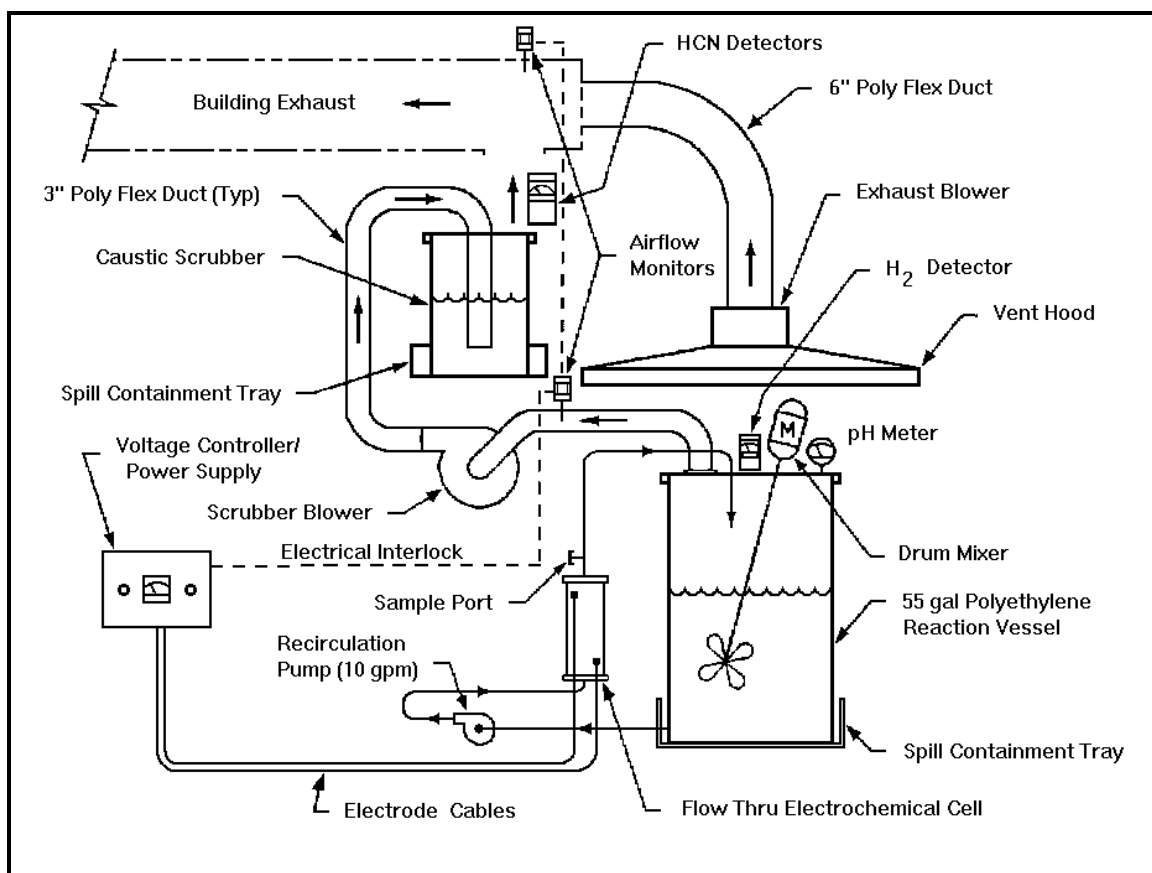


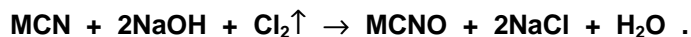
Figure 1. Process schematic and engineering drawing.

Process Description

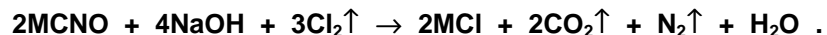
Sodium chloride (NaCl) is added to the cyanide waste solution as the chlorine source. The waste is circulated through an electrolytic cell equipped with inert platinum anodes. The electrodes generate powerful oxidizing agents, such as chlorine gas and hydroxyl radicals, and convert the cyanide to carbon dioxide and nitrogen. Under high current, the 1–5 percent sodium chloride solution is broken down according to the following reaction:



The chlorine gas generated in situ oxidizes the cyanide species to form cyanates (MCNO, where M is a metal such as sodium, cadmium, copper, or gold) according to the following equation:



The continuous production of sodium hydroxide (NaOH) keeps the pH elevated and prevents the formation of toxic cyanogen chloride gas (CNCl). Chlorine gas further oxidizes the MCNO to carbon dioxide (CO₂) and nitrogen (N₂) according to the following equation:



Cyanide in solution readily forms complexes with heavy metals, some of which are more difficult to destroy by chlorination. Cyanide forms readily destroyed by chlorination are referred to as amenable cyanide. RCRA Universal Treatment Standards include concentration levels for both total cyanide and amenable cyanide.

Destruction time depends on the type of cyanide and the concentration. Generally, 10 ppm chlorine will destroy 1 ppm cyanide. Although sodium hydroxide is generated in the cell, the pH in the reaction vessel is continuously monitored to ensure safety and cyanide destruction. Operating experience has shown that periodic additions of sodium (or potassium) hydroxide are required to supplement that generated in the cell to maintain pH levels at 10 or above.

In the RFETS demonstration, after destruction of the cyanides, the residual solution typically contained hazardous metals and was sent to the site's liquid waste treatment facility for final immobilization treatment.



SECTION 3

Demonstration Plan

Technology performance was assessed using information from the treatability study and subsequent waste treatment operations at RFETS. The effectiveness of the electrochemical chlorination process was demonstrated in the treatability study using four different types of cyanide waste:

- Study 1: cadmium cyanide plating bath
- Study 2: gold cyanide plating bath
- Study 3: dilute cyanide plating rinse
- Study 4: cyanide chemical wastes, e.g., potassium cyanide, silver electrolyte solution (AgCN), and hydrogen cyanide detection calibration gas

The first activity in the study was to characterize the selected excess cyanide wastes and chemicals for various parameters, including pH, total cyanide, amenable cyanide, gross alpha and beta activity, total uranium, and metals. Characterization provided information on the nature of the waste so that the process could be designed and operated safely and effectively. In addition, this information served as a baseline (in addition to the Universal Treatment Standards) against which results were compared.

For treatability studies 1–3, the system was charged by pumping up to 190 L of cyanide waste solution into the reaction vessel. The system was allowed to operate continuously until the desired cyanide level was reached. Samples were collected periodically and analyzed to monitor cyanide destruction. The excess cyanide chemicals for treatability study 4 were first dissolved in 100 L of caustic solution and then treated. The resulting cyanide-free waste still contained hazardous metals and was disposed of in the process waste drain for final immobilization treatment.

As part of the treatability study, a comprehensive analysis was performed on the remaining drums of waste cyanide solutions. Six additional drums were determined to require treatment for cyanides. These wastes were treated following approval of a RCRA Part B permit modification by the state of Colorado.

Treatment Performance

Results of the RFETS tests on four different types of cyanide wastes are summarized in Table 1. Three of the four treatability tests successfully destroyed cyanides to levels below the RCRA Universal Treatment Standards for total cyanide (590 mg/kg) and amenable cyanide (30 mg/kg). The fourth test was terminated due to time constraints before the amenable cyanide was reduced to levels below the regulatory standard. Over 800 L of waste was treated in this phase.

- The waste cadmium cyanide solution had an initial total cyanide concentration of 11,000–13,600 mg/L (based on results reported by four laboratories). After treatment, the final total cyanide concentration was substantially below the treatment standard of 590 mg/kg for F007, spent cyanide plating bath solutions from electroplating operations. Due to time constraints, the cadmium cyanide test was terminated before the amenable cyanide was reduced to ≤ 30 mg/kg. Given sufficient time, this standard would have been achieved, as proven by subsequent studies.
- Initial analysis of the gold cyanide solution resulted in a total cyanide concentration of 1,200–2,500 mg/L. In addition, the solution was found to contain 13 g of gold per liter. After treatment, the total cyanide concentrations were 17–55 mg/kg, and amenable cyanide ranged < 5 –18 mg/kg. Both total and amenable cyanide concentrations were substantially below treatment standards. Approximately 1,800 g of metallic gold was recovered during the process and returned to the site's precious metal inventory.



Table 1. Summary of treatability study results

Waste type	Mean initial cyanide concentration ^a (mg/L)		Mean final cyanide concentration ^a (mg/L)		Universal Treatment Standard (mg/kg)	
	Total	Amenable	Total	Amenable	Total	Amenable
Cadmium cyanide plating waste	12,350	13,600 ^b	108	104.5	590	30
Gold cyanide plating waste	1,850	710	36	18 ^b	590	30
Dilute cyanide plating waste	170 ^b	65 ^b	<2.5	No data ^c	590	30
Hydrogen cyanide gas	36 ppm		<2.5	No data ^c	590	30
Potassium cyanide reagent	2,070 ^d		<0.1	No data ^c	590	30
Silver electrolyte solution	No data ^e		<30	No data ^c	590	30

^aWaste samples were analyzed by up to four different laboratories. For the purpose of summarizing the data in this report, mean values of laboratory results were calculated from reported data. Detailed analytical results are given in C. M. Jensen and D. M. Stull, *Electrochemical Chlorination for Destruction of Cyanide Wastes, End-of-Year Test Report*, Internal Report No. TI95-014, Kaiser-Hill Company, 1995.

^bBased on single reported value (the concentration of amenable cyanide cannot be physically greater than the total concentration).

^cAmenable cyanide was not analyzed because the total cyanide value was less than the 30 mg/kg Universal Treatment Standard for amenable cyanide.

^dInitial sample from treatment process.

^eNo initial concentration was reported due to an unknown interference.

Six additional drums (over 1,130 L) of spent cyanide plating wastes were successfully treated under a RCRA Part B permit modification at RFETS. Table 2 summarizes the results of this treatment.

Table 2. Summary of RCRA Part B permit operation results

Drum number	Initial cyanide concentration (mg/kg)		Final cyanide concentration (mg/L)		Universal Treatment Standard (mg/kg)	
	Total	Amenable	Total	Amenable	Total	Amenable
N00049	169	103	25	NA	590	30
N00045	279	279	17	NA	590	30
N00050	360	331	18	NA	590	30
N00048	1,600	653	104	27	590	30
R00433	2,400	26	<0.01	<0.01	590	30
R00347	1,700	828	85	<1	590	30

Key System Parameters

The key system parameters, refined during the permit application process and actual operation of the treatment unit, include the following:

- Maintenance of pH and ventilation airflow are the primary safety mechanisms for this system. Therefore, for safety as well as proper operation, the following parameters were set:
 - pH of solution in reaction vessel ≥ 10
 - pH of scrubber solution ≥ 12
 - ventilation airflow maintained through both the reaction vessel and the vent hood

Maintenance of pH prevents the formation of hazardous hydrogen cyanide gas. Because experience shows that changes happen slowly, monitoring and adjustment of pH are manual operations.

Maintaining airflow prevents buildup of hydrogen gas (an operational byproduct of the electrolytic



cell) and other hazardous gases in the event of system upset. Airflow is constantly monitored in both the scrubber ventilation line and the vent hood. Loss of ventilation shuts the system down automatically.

- Maintaining recirculation pump flow and system conductivity are other key parameters for proper operation as follows:
 - 50 A at <10 V to the electrochemical cell
 - 10-gpm flow through the recirculation pump

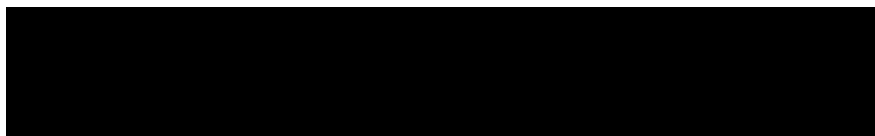
Electrical current through the cell is controlled primarily by adding sodium chloride to make the solution conductive. Secondly, the power supply is adjustable for current output. The power input is also adjusted if necessary to keep the temperature of the solution below 50°C. Higher temperatures cause condensation problems in the ventilation system. A nonadjustable 10-gpm pump provides recirculation. This pump is small enough to allow sufficient residence time for the solution in the cell and large enough to provide thorough mixing of the solution in the vessel.

Limitations/Potential Problems

- Iron cyanides are very stable compounds and may therefore be considered a minimal hazard. However, these are cyanide complexes and must be destroyed prior to disposal. Although it has not yet been proven, the plan at RFETS was to destroy these compounds using a very aggressive hot acid to form hydrogen cyanide in a closed system. The hydrogen cyanide would then be captured and destroyed in a basic chlorine solution. Due to the very small quantity of iron cyanides at RFETS, a bench-top process using hypochlorite solution was planned. The scale of the electrochemical chlorination system would be too large for this small requirement.
- RFETS encountered several interferences during the development of the electrochemical chlorination process. Ammonia and organic compounds were problems in several batches. The chlorination process reacts more favorably with these constituents than with the cyanide contaminants. As a result, the destruction process is slowed in proportion to the quantity of the interfering materials in solution.



SECTION 4



Competing Technologies

Alkaline chlorination is the most widely used cyanide destruction method. The process uses chlorine gas in high-pH solutions to chemically destroy cyanide complexes. The electrochemical chlorination method is a safer alternative method of cyanide destruction because it does not require an external source of chlorine gas.

Technology Applicability

- The electrochemical chlorination cyanide destruction process has been demonstrated to effectively treat solutions containing cyanide salts. These solutions may be spent wastes from plating processes or from other sources of cyanide salts.
- The electrochemical chlorination process destroys cyanide and removes heavy metals as a hydroxide precipitate.
- Electrochemical cells can treat cyanide solutions ranging from weak rinses (3 ppm cyanide) to plating baths (20 g/L). Cells can be purchased for handling small (50-gal) batches up to 2,000-gpm continuous flow.
- Silver may interfere with current analytical methods.

Technology Maturity

The electrochemical chlorination technology has been successfully demonstrated at RFETS in both treatability studies and RCRA Part B-permitted operations. As an application of commercially available equipment, the technology is mature and available for widespread use, as witnessed by its use to completely eliminate a low-level mixed waste from the RFETS inventory. Further development to reduce the cost of labor may be appropriate in the areas of process monitoring and automatic process control.



SECTION 5

Methodology

The following cost estimates are based on the actual cost for treatment of six drums of cyanide wastes under the RFETS RCRA Part B permit modification (second phase of treatment) approved in March 1996. These estimated costs reflect a 55-gal batch system.

Cost Analysis and Conclusions

- Capital (one-time) costs

Equipment	\$20,000–\$30,000
RCRA Part B permit modification	120 hours labor

- Operating costs

Supplies (caustic, salt, etc.)	\$100/batch
Operator	half-time for three shifts during operation
Management	one-quarter time for one shift during operation

The Part B permit approved by the state of Colorado required hourly inspection of the treatment unit during operation. Installation of additional monitoring instrumentation could reduce inspection frequency and operational costs.

No cost-benefit information is available on this technology. Industry with similar (nonradioactive) waste types could contract treatment and disposal through a waste management vendor for \$200 to \$500 per drum, depending on the specific chemical and physical characteristics of the waste. That alternative was not available for the wastes in this demonstration because of their classification as mixed waste.



SECTION 6

Regulatory Considerations

- **National Environmental Policy Act.** A Categorical Exclusion was applied to this action in accordance with 10 CFR Section 1021, Subpart D, Appendix B6.1: Removal actions under CERCLA [Comprehensive Environmental Restoration, Compensation, and Liability Act] and removal type actions similar in scope under RCRA and other authorities which meet the CERCLA regulatory cost and time limits or exemptions.
- **Resource Conservation and Recovery Act.** A testing facility conducting a treatability study is not subject to further requirements of hazardous waste regulations provided conditions in 40 CFR Section 261.4(f)(1–11) are met. A treatability study exemption was granted for the cyanide destruction study.

Following successful treatment of waste under the treatability study exemption, RFETS requested a RCRA Part B permit modification. This action was based on plans to treat the entire inventory of cyanide-bearing waste plating solutions using the electrochemical chlorination cyanide destruction process. In March 1996, the Colorado Department of Public Health and Environment approved the request.

Alkaline chlorination treatment technologies such as electrochemical chlorination were formerly the RCRA Best Demonstrated Available Technology for treatment of listed cyanide wastes (F007). Recent changes to RCRA regulations have adopted concentration-based treatment standards for these wastes (590 ppm total cyanide and 30 ppm amenable cyanide).

- **Clean Air Act.** Colorado Air Quality Control Commission (CAQCC) Regulation No. 3 regulates nonradionuclide air pollutant emissions for all Colorado industries. The provisions of the regulation identify criteria and hazardous air pollutants, establish air inventory reporting thresholds for regulated air pollutants, and set forth construction and operating permit application guidelines. An air emissions evaluation was conducted to determine the emissions inventory reporting and permit requirements for the cyanide destruction project. The production and evolution of chlorine gas and hydrogen cyanide were identified as the only potential nonradionuclide air pollutant emissions from the project. An assessment determined that the project was not subject to any air emission or air permitting requirements under CAQCC regulations.

A radionuclide air emission assessment was also performed to determine potential impacts under 40 CFR 61, Subpart H. The analysis did show the presence of low-level beta emissions attributed to potassium-40. No plutonium or uranium was present in the solutions, so there were no radionuclide air quality impacts.

Safety, Risks, Benefits, and Community Reaction

- Worker Safety

Chemical Hazards. Cyanogen chloride (CNCl), hydrogen cyanide (HCN), chlorine gas (Cl₂), and the caustic solutions in the reaction vessel and caustic scrubber represent the principal chemical hazards. The process is designed to maintain a pH >10 in the reaction vessel and ≥12 in the caustic scrubber to prevent the evolution of these hazardous gases. The ventilation blower maintains airflow through the reaction vessel and to the caustic scrubber. This airflow is the primary safety system. If the process gets out of tolerance and evolves any cyanogen chloride or hydrogen cyanide gas, the airflow carries all gases from the reaction container to the caustic scrubber container. High-pH liquids quickly capture any cyanogen chloride or hydrogen cyanide gases. In addition to the airflow, the



system includes a hydrogen cyanide detector near the caustic scrubber, set to produce an audible alarm before there is a health hazard.

Spills, releases, and/or chemical burns are additional hazards of the system. These hazards are controlled by limiting the maximum pressures and flow rates used in the system, using containment trays under the reaction vessel and caustic scrubber system, using appropriate industry standard personal protective equipment, and ensuring proper training of operational and management personnel.

Explosive Hazards. The scenario of highest risk is the potential of a fire or explosion resulting from hydrogen gas buildup. The explosive concentration range of hydrogen gas is 4 percent to 95 percent, so it is necessary to control potential accumulation. In the RFETS setup, control is accomplished by proper airflow and ventilation. A hydrogen detector at the reaction vessel is set to produce an audible alarm before there is a fire hazard.

Electrical Hazards. The electrolytic cell reduces the incoming 120-V AC power supply to less than 10 V. All other equipment associated with the system is either battery operated or powered by 110 V AC. All electrical equipment is National Electrical Code certified. Electrical supply failures to the process result in a safe system shutdown.

Other Hazards. The standard fire, mechanical, and electrical hazards associated with any industry must also be addressed for safe implementation of this technology.

- Community Safety

Proper mitigation of worker safety issues will take care of any community safety concerns.

- Environmental Impacts

Proper mitigation of worker safety issues will also minimize the potential for environmental impacts.

- Community Perception

All contacts with representatives of the various stakeholders in the RFETS demonstration were positive. Briefings on the status of the technology were presented at several points during the project. At the completion of the project, three separate presentations were held, one at an international conference. The response from all these meetings was positive, with most commentors expressing relief that a waste as hazardous as cyanide was being singled out for destruction and removal from the site's inventory.



SECTION 7

Technology Selection Considerations

- The electrochemical chlorination process works only on water-soluble waste.
- Very stable cyanide complexes (e.g., iron cyanides) are very difficult to destroy by chlorination and would not be appropriate for treatment using this technology.
- The waste treated at RFETS was mixed; however, it was only “radioactive by association” due to the building in which it was generated. Waste containing relatively high levels of radioactivity could be treated by this technology, but other component considerations would be in order.

Design Issues

- Because of budget limitations, the RFETS system was a minimal system assembled from scavenged components. As a result, it had questionable reliability and required too much operator attention. Ideally, electrochemical chlorination system components would be selected and installed in a permanent, reliable manner.
- The system should be designed for easy cleaning during batch change. Operation of the system causes precipitate to collect in the reaction vessel and electrochemical cell. These components should be easily dismantled for cleaning and sludge removal.

Implementation Considerations

- Project staff should work closely with the regulators during implementation. RFETS’ good relationship with the state of Colorado was an important factor in the success of the project, especially during the permitting phase.
- The permit should be negotiated to be as general as possible, especially in reference to location of the equipment and specific components used. RFETS is in the unique position of dismantling the site. The building where the cyanide treatment system is located is slated for decontamination and decommissioning in the near future, and relocation will require a modification to the permit. In addition, the permit is so specific that updating the system without modifying the permit may become a problem.

Technology Limitations and Needs for Future Development

The basic electrochemical chlorination system is an adaptation of well-known technology. The only development that may be appropriate would be for additional instrumentation and automated controls to reduce the frequency of inspections and maintenance.



APPENDIX A



Jensen, C. M., and D. M. Stull 1995. *Electrochemical Chlorination for Destruction of Cyanide Wastes, End-of-Year Test Report*. Internal Report No. TI95-014, Kaiser-Hill Company.

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